Supercond. Sci. Technol. **20** (2007) L59–L62 doi:10.1088/0953-2048/20/10/L02

RAPID COMMUNICATION

Proof-of-principle experiments for react–wind–sinter manufacturing of $Bi_2Sr_2CaCu_2O_x$ magnets

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Received 22 July 2007, in final form 15 August 2007
Published 4 September 2007
Online at stacks.iop.org/SUST/20/L59

Abstract
The manufacturing of long lengths of $Bi_2Sr_2CaCu_2O_x$ (Bi2212) wire has led to the development of high-field magnets at low temperature. Both react-and-wind and wind-and-react manufacturing of magnets, however, have significant drawbacks that limit the performance of the conductor in magnet form. In this letter, an alternative approach to magnet manufacturing, react–wind–sinter, is proposed to avoid these drawbacks by splitting the heat treatment into two separate stages, with the temperature/time-sensitive peritectic melting occurring before magnet winding and the remaining solid-state sintering after magnet winding. Results from proof-of-principle experiments show that the split heat treatment does not adversely affect the conductor performance and that the subsequent heat treatment is capable of preventing bending-induced degradation of the conductor if the heat treatment is interrupted just after re-solidification from the peritectic melt.

1. Introduction

High-temperature superconductors (HTS) have advanced significantly and there are now three materials for which industry is manufacturing sufficient lengths of conductor to construct magnets: $YBa_2Cu_3O_{7-z}$, $Bi_2Sr_2Ca_2Cu_3O_y$, and $Bi_2Sr_2CaCu_2O_x$ (Bi2212). As a result, the number and variety of magnet applications is growing and includes magnets for motors, fault current limits, generators, medicine and scientific applications [1–5]. Magnet applications can be categorized into three regimes according to the magnetic field and operating temperature of the magnet. The operating regime is dictated in part by the requirements of the application, and is coupled directly to the selection of the appropriate magnet conductor. These regimes include high temperature–low magnetic field, intermediate temperature and magnetic field, and low temperature–high magnetic field. Note that the low temperature–low magnetic field regime is fulfilled by NbTi and Nb_3Sn, and the high temperature–high magnetic field regime remains unreachable.

At low temperature and high field, Bi2212 has the highest engineering critical current density (J_e) and is thus the preferred conductor for high-field solenoids [6–8] and potentially for high-field racetrack magnets for accelerators. Bi2212 is also the only HTS conductor with high J_e in a round wire, furthering its attractiveness for high-field magnets. In general, high-field magnets have large Lorentz forces and thus the dependence of J_e upon strain and the conductor modulus are important for magnet design and performance. Bi2212 conductors are co-processed with Ag and Ag-alloy matrices, which dominate the mechanical properties. Furthermore, Bi2212 itself is a brittle ceramic. As a result, Bi2212 is highly strain sensitive [9].

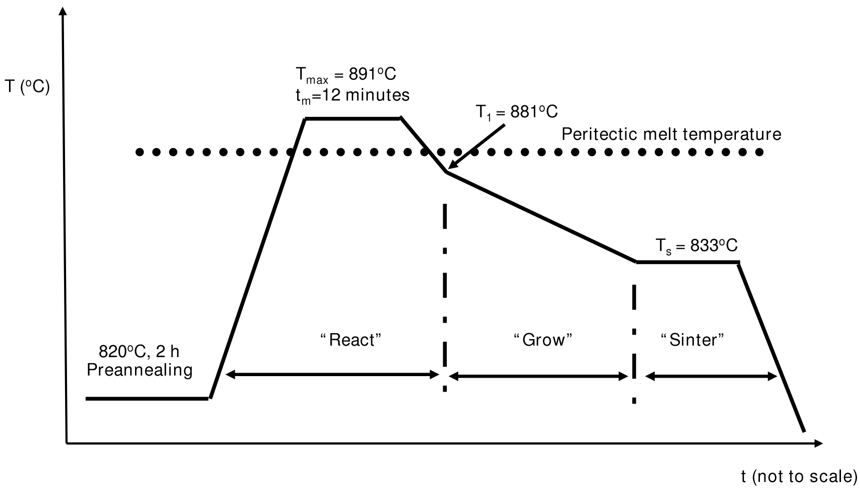

Figure 1. Conventional heat treatment process for Bi2212 wires.

Bi2212 conductors only have high J_e when processed in flowing oxygen above the peritectic melting temperature. A typical temperature–time profile is shown in figure 1. The profile can be summarized as having three parts: the partial-melt reaction ('React') which includes heating from room temperature above the peritectic temperature and then re-solidifying, the growth step ('Grow'), during which the conductor is slowly cooled within the solid Bi2212 phase field, and the sintering step ('Sinter') during which the sample is held at a constant temperature until cooling back to room temperature.

Traditionally, there are two approaches for manufacturing superconducting magnets: react-and-wind (R&W) and wind-and-react (W&R). In R&W, the conductor is fully processed and then wound into the desired magnet geometry. Wire and magnet manufacturing are decoupled, but any strain associated with the magnet construction must be included in the overall mechanical analysis of the magnet. The insulation and other materials are incorporated after the conductor is fully processed, so materials compatibility issues are limited to those associated with differential thermal contraction during cool-down. In W&R, the conductor is wound into the final magnet geometry before it has been heat treated to form the superconducting phase. In this case, all materials within the magnet experience the conductor heat treatment conditions. Because the superconducting phase is formed in its final geometry, there is no remnant strain on the conductor associated with winding.

Both R&W and W&R have been used for the manufacturing of Bi2212 magnets, but both approaches have significant problems, particularly for high-field magnets. Although there has been success for Bi2212 R&W tape magnets, the highest $J_e(B)$ is available in round wires, which are more susceptible to bending strain. As conductor and magnet performance improve, the Lorentz force increases and the Bi2212 strain limit becomes prohibitive. To avoid these mechanical problems, the recent focus has been on W&R magnets. In this case, the primary challenges are associated with the oxygen heat treatment. The presence of pure oxygen at nearly 900 °C severely limits options for insulation and reinforcement. The conductor performance is sensitive to both the peak temperature and the time at the peak temperature, such that the available processing window is only a few degrees celsius. For short conductors and small magnets this is not problematic. For larger magnets, however, thermal diffusion and oxygen diffusion can both limit the uniformity of the magnet performance. Furthermore, it is not uncommon for the Bi2212 to leak from the conductor and interact with the insulation when heat treating W&R magnets.

In this letter an alternative approach for manufacturing Bi2212 is explored and proof-of-principle experiments on short samples are presented. In this 'react–wind–sinter' (RWS) approach, the heat treatment is split into two heat treatments separated by a cool-down to room temperature, as shown in figure 2. This concept was originally proposed in [10], but in that case the reaction step was performed using a continuous process where the conductor was pulled through a furnace with a temperature profile intended to emulate the required temperature–time conditions. As a result of this complicating aspect, and the relative immaturity of Bi2212 conductor at that time, those results were inconclusive. For the manufacturing approach proposed here, the conductor will go through the reaction stage before winding using a conventionally controlled furnace, be cooled to room temperature, wound into the final magnet geometry, and then re-heated to complete the full heat treatment process. Thus, the partial-melt reaction, to which the performance is particularly sensitive, is experienced by bare conductor (like R&W), but the conductor experiences a lengthy heat treatment after winding to relax the bending stress (like W&R). Proof-of-principle experiments focus on two essential questions:

- Can high J_e be obtained in conductors heat treated by the split process shown in figure 2?
- Does bending between the partial-melt reaction and the solid-state sintering affect J_e?

2. Experimental approach

Multi-filamentary Bi2212 round wires (0.81 mm diameter) were manufactured by Oxford Superconducting Technology, as

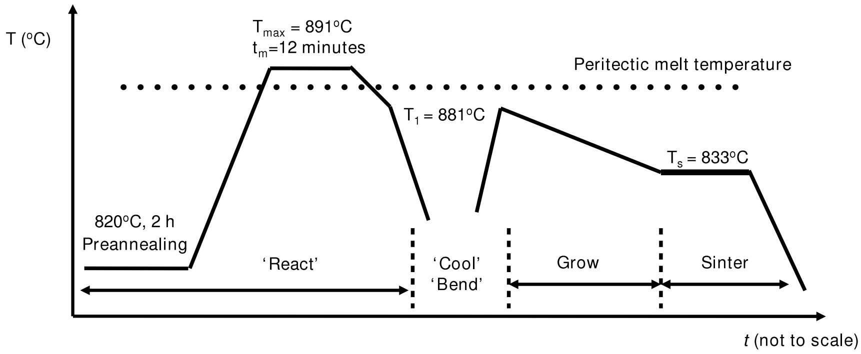

Figure 2. An example of a react–wind–sinter heat treatment. Illustrated is 'react–cool–bend–grow–sinter' (RCBGS). Variations include 'react–grow–cool–bend–sinter' (RGCBS), depending on the specific sequence of the heat treatments.

Figure 3. Macor sample holder (disassembled and assembled) for heat treating Bi2212 wires after bending.

(This figure is in colour only in the electronic version)

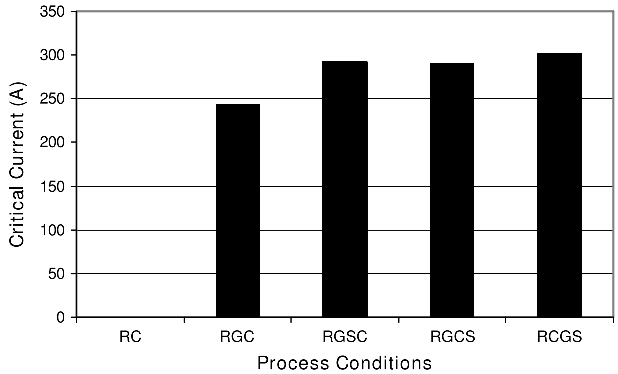

Figure 4. Critical current at 4.2 K, 5 T, for Bi2212 wires that have been processed with variations of the RWS heat treatment shown in figure 2. It is seen that the split heat treatments do not reduce the wire performance.

described in [11]. All samples were from a single spool of wire. All heat treatments were performed in flowing oxygen in a tube furnace. Sample processing steps, which are seen in figures 1 and 2, are react 'R', grow 'G', sinter 'S', and cooling to room temperature 'C'. The peak temperature during processing and all ramp-rates and hold times are identical for each sample; the only variations between samples are if/when the process is split and whether or not the sample is bent ('B'). Samples are named for their specific process and include: RGSC (conventional heat treatment shown in figure 1), RC, RGC, RGSC, RCGS, RGCS, RCBGS, RGCB and RGCBS.

The bending ('B') step is intended to emulate the winding of a magnet. In these experiments, bending was accomplished by slowly forcing the wire into the macor sample holder shown in figure 3. The sample holder has slots with bend diameters ranging from 40 to 100 mm. A separate slot for straight (infinite diameter) wires is also included. After inserting samples into the holder, the assembly was inserted into the furnace to complete processing. The apparatus was designed such that disassembly after heat treatment would be easy and not risk damaging the samples after heat treatment.

Critical current (I_c) measurements were performed using the four-probe method in liquid helium (4.2 K) in a 5 T magnetic field. An electric field criterion of 1 μV cm^{-1} was used to determine I_c.

3. Results

Figure 4 shows $I_c(5\ \mathrm{T}, 4.2\ \mathrm{K})$ for RC, RGC, RGSC, RGCS and RCGS samples. Note that 'RGSC' is the conventional heat treatment shown in figure 1. Each data point is the average of at least four samples. The RC samples are resistive, confirming that the growth and sinter steps are necessary. The RGC samples have an average I_c that is about 80% of the fully processed samples. The RGSC, RGCS and RCGS results are all within about 3% of each other, clearly showing that splitting the process into two separate heat treatments is not detrimental.

Figure 5 shows $I_c(5\ \mathrm{T}, 4.2\ \mathrm{K})$ for RCBGS, RGCB and RGCBS versus the inverse of the bending diameter, which is proportional to the bending strain ($1/d = 0$ represents straight samples). Again, each point is the average of multiple samples. The performance of the RCBGS samples is independent of the bending, while that of the RGCB and RGCBS are strongly affected by bending, with about a 50% decrease in current for $1/d = 10$ m^{-1} (a 100 mm bending diameter), and a slow decrease in current with additional bending. These results show that the combination of the growth and sinter steps is effective in healing damage to the Bi2212 caused by the bending, but that the sintering step alone is not. The sintering step does, however, improve the transport in unbent wires.

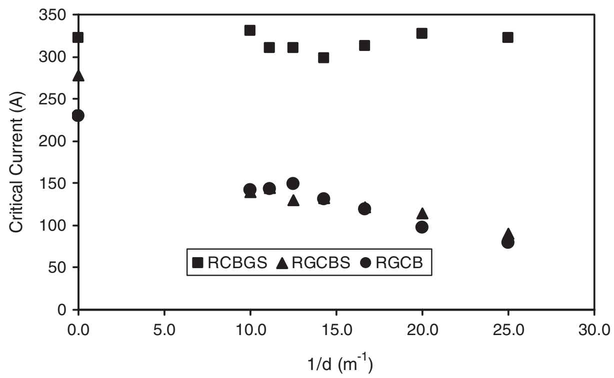

Figure 5. Critical current at 4.2 K, 5 T, versus the inverse of the bending diameter for RCBGS, RGCB and RGCBS wires. For constant wire diameter, the inverse of the bending diameter corresponds to bending strain. It is seen that that the combination of the growth and sinter steps is effective in healing damage to the Bi2212 caused by the bending, but that the sintering step alone is not.

4. Conclusions

A new approach to manufacturing Bi2212 magnets which avoids the primary problems of R&W and W&R is proposed. This approach splits the heat treatment into two steps, the first of which is performed before winding the magnet that includes the requisite peritectic melting and the latter which includes solid-state sintering. Proof-of-principle experiments presented here show that the split heat treatment does not adversely affect the conductor performance and that the subsequent heat treatment is capable of preventing bending-induced degradation of the conductor if the heat treatment is interrupted just after completing the reaction step.

Acknowledgments

The authors thank Oxford Superconducting Technology for providing the conductor, and Timothy Effio, Xiaotao Liu, Manuel Ramos, Tengming Shen and Ulf Trociewitz for assistance with transport measurements and many useful discussions. Tengming Shen is also thanked for assistance in drawing figures 1 and 2.

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